Optimization of Osmotic Dehydration Process of Grapes Using Response Surface Methodology

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Abstract

Response surface methodology was used for quantitative investigation on water and solids transfer during the osmotic dehydration process of the grapes in sucrose solution using Box-Behnken experimental design. Effects of temperature (35–55^{0}C), sucrose concentration (40–60^{0}Brix) and processing time (100–200 min)), on osmotic dehydration of grapes were estimated. Quadratic regression equations describing the effects of these factors on the water loss, solids gain, rehydration ratio and sensory score were developed. It was found that effects of concentration and temperature were more significant on the water loss than that of processing time. As for solids gain processing time and temperature were the most significant factors. The osmotic dehydration process was optimized for water loss, solute gain, rehydration ratio and sensory score. Optimum conditions obtained by numerical optimization were temperature -36.92^{0}C, processing time -160.57 minutes and sucrose concentration - 60^{0}Brix solution to achieve maximum water loss, rehydration ratio and sensory score, and lower solute gain. Corresponding to these optimum conditions, the predicted value for water loss was 40.54 (g/100 g initial sample), 10.06 solid gain (g/100 g initial sample), 3.05 rehydration ratio and 7.63 sensory score.

Keywords

Osmotic Dehydration; Optimization; Response Surface Methodology

Introduction

Grape (*Vitis vinifera*) , one of the most popular and palatable fruits in the world . is basically a sub-tropical crop, which is also good source of vitamin C, vitamin A, vitamin K, carotenes, β-complex vitamins such as pyridoxine, riboflavin, and thiamine. Grape containing minerals like: folate, calcium, chlorine, copper, fluorine, iron, magnesium, manganese, phosphorus, potassium, silicon and sulfur in abundance, is edible without processing or used for making jam, juice, jelly, wine, grape seed extracts, raisins, vinegar, and grape seed oil. Raisin , one of the most important dried products obtained by drying of grapes, are directly used as ingredients in the confectionery and in the form of raisin paste applied to fillings, baked goods, sauces, microwavable coating and also for natural coloring of other food products (Veronique and David 1993). The preservation of grapes by drying is used in many industries in the world, where grapes are cultivated. Drying of grapes, either by sun drying, shade drying, or hot air drying produces raisins (Pangavhane and Sawhney 2002). Grapes are considered to be rather complex system with an outer waxy cuticle and pulpous material inside. During drying of the grapes, the waxy cuticle is main obstacle which restricts and controls the moisture diffusion in the grapes (Grnearevic and Radler 1971). Di Matteo et al (2000) proposed a physical treatmentbased on the superficial abrasion of the grape peel, which was found to be as effective as the traditional chemical dipping method in reduction of drying times.

Osmotic dehydration is a technique that involves product immersion in a hypertonic aqueous solution leading to loss of water through the cell membranes of the product and subsequent flow along the inter-cellular space before diffusing into the solution (Sereno et al 2001). Two major simultaneous counter-current flows occur during osmotic dehydration: water flow out of the food into the solution and a simultaneous transfer of solute from the solution into the food (Madamba 2003). Osmotic dehydration,

which is effective even at ambient temperature and protects the colour, flavour and texture of food from heat, is used as a pretreatment to improve the nutritional, sensorial and functional properties of food. Generally osmotic concentration would not give low moisture content to be stored for long time. Osmo-dried products should be processed further (air drying, vacuum drying etc.) to obtain shelf-stable products (Pointing, 1973). The osmo dried papaya and mango slices were dried in a cabinet dryer at 60^{0}C for 6 hrs to obtain 16 per cent moisture content (Gurumeenakshi et al 2005). Shedame and Patil (2009) studided osmotic dehydration grapes for raisin preparation for long immersion period (6-8 hr) followed by tray drying. In recent years, osmotic dehydration has received considerable attention due to the low temperature employed in the process that improves the final product quality and reduces energy consumption. Some researchers have also tried to increase the rate of osmotic mass transfer to reduce the processing time (Ispir and Togrul 2009, Mundada et al 2011).

RSM is a collection of statistical techniques to design experiments, build models, evaluate the effects of factors and search the optimum conditions, in which, several factors are simultaneously varied. The multivariate approach which reduces the number of experiments, improves statistical interpretation possibilities, and evaluates the relative significance of several affecting factors even in the presence of complex interactions, is employed for multiple regression analysis using quantitative data obtained from properly designed experiments to solve multivariable equations simultaneously. It is widely used for multivariable optimization studies in several biotechnological processes such as optimization of media, process conditions, catalyzed reaction conditions, oxidation production, fermentation, biosorption of metals etc. (Chang et al 2006, Soo et al 2004, Wang and Lu 2005) as well as has been used to determine the optimal values for process parameters in various processes (Harris et al 1990, Mannan et al 2007). Hence, the purpose of this study is to optimize the osmotic dehydration of seedless grapes using sucrose solution by Response Surface Methodology (RSM).

Materials and Methods

Preparation of Samples and Osmotic Dehydration

Fresh ripe commercially harvested Indian Thompson seedless grapes were procured from local market on daily basis prior to each set of experiments. After washing, gapes were blanched at 70^{0}C due to the loss of outer waxy cuticle layer and then grape peel was done manually to remove the outer waxy cuticle, which was found to be as effective as the traditional chemical dipping method in reduction of drying times (Di Matteo et al. 2000). Grape slices of approximately 1.5 cm thickness and 1.75 cm diameter were cut carefully with the help of razor blade. For each experiment 50 gram of grapes were put in a conical flask containing calculated volumes of osmotic solutions of different (40 – 60^{0}B) concentrations preset at the desired temperatures (35–55^{0}C) in incubator shaker. Stir was used to reduce the mass transfer resistance at the surface of the grapes and to ensure good mixing and close temperature uniformity as well as control in the osmotic medium (Chopra 2001, Mavroudis et al 1998). The sample to solution ratio was kept at 1:5 by weight (Azoubel and Murr 2004, Kar and Gupta 2001, Pokharkar and Prasad, 2002).

At specified time intervals that is 100 min, 150min and 200min, grapes were removed from the osmotic solutions and rinsed with water to remove surplus solution adhering to the surfaces. These osmotically dehydrated slices were then spread onto absorbent paper to remove free water present on the surface. 10 gm of the product was used for determination of dry matter by oven-drying. The remaining part of each product sample was dried to a final moisture content of 6% (wet basis) using a hot air drier preset at 60^0 C air temperature and 1.6 m/s air velocity. The dried samples were cooled in desiccators containing silica gel for 1 h, packed in HDPE (high density polyethylene) bags, and kept at ambient temperature for quality analysis.

Experimental Design and Statistical Analysis

Response surface methodology (RSM) was used to estimate the effects of osmotic dehydration process on water loss, solid gain, rehydration ratio and sensory score. A Box-Behnken experimental design with three blocks and three replicates was used with temperature (35–55^{0}C), sucrose content (40–60^{0}B) and processing time (100– 200 min) being the independent process variables. Level of input variables in coded and uncoded form is given in Tabe1. Statistical analysis Response Surface Methodology was used to model and optimize selected response variables. The statistical software package (Design-Expert version 8.0.6, Stat-Ease Inc.Minneapolis, USA, trial version) was used for regression analysis of experimental data

and to plot response surface. The generalized second-order polynomial model was used in the response surface analysis. Experimental data were fitted to the second order polynomial model and regression coefficients obtained. The model was simplified by dropping terms which were statistically insignificant (p>0.05) by means of analysis of variance (ANOVA). The response surface and contour plots were generated by a variable constant in the second-order polynomial model. Optimization of independent variables for osmotic dehydration of grapes was determined by superimposing the plots for selected responses. The optimum conditions and the predicted values of the response variables were obtained using the Design Expert software.

Water Loss (WL) and Solute Gain (SG) During Osmotic Dehydration

The water loss and solute gain during osmotic dehydration were calculated by the following equations (Ozen et al 2002):

$$\text{Water loss/100g of fr esh fruit} = \frac{(W_o - W_t) + (S_o + S_t)}{W_o} \text{ X } 100 \quad (1)$$

$$\text{Solid gain/100g of fresh fruit} = \frac{S_t - S_o}{W_0} \text{ X } 100 \quad (2)$$

where W_o is the initial weight of fruit (g), W_t is the weight of fruit after osmotic dehydration for any time t (g), *So* is the initial weight of solids (dry matter) in the fruit (g), and *St* is the weight of solids (dry matter) of fruit after osmotic dehydration for time t (g).

Rehydration Ratio (RR)

The rehydration of dried grapes slices was determined by soaking a known weight of each sample in a sufficient volume of water (approximately 30 times the weight of dried grapes) at room temperature. At the end of the rehydration period, i.e. 10–12 h, which was found to be adequate for the grapes to reach a constant weight, the slices were weighed after the removal of excess water with the help of absorbent paper. The rehydration ratio was computed as:

$$\text{Rehydration ratio} = \frac{\text{Weight of rehydrated grapes (g)}}{\text{Weight of dehydrated grapes (g)}} \quad (3)$$

Sensory Evaluation of Rehydrated Grapes (SS)

Organoleptic quality of rehydrated grapes was determined with the help of a 10-member consumer panel, using a 9- point hedonic scale, following standard procedure. The aspects considered for rehydrated grapes were colour, appearance, taste, favour, and overall acceptability. The average scores of all the 10 panellists were computed for different characteristics.

Results and Discussion

Fitting Models

Experiments were performed according to the Box behkan experimental design given in Table 2 in order to search for the optimum combination of parameters for the osmotic dehydration of grapes. A Model F-value of 53.82, 23.13, 4.89, and 13.45 for WL, SG, RR and SS implies respectively that the model is significant. The Lack of Fit F value of 2355.82, 197.31, 1319.2, and 36.25 for WL, SG, RR and SS implies the Lack of Fit is significant. The Fisher F-test with a very low probability value ($P_{model} > F = 0.05$) demonstrates a very high significance for the regression model. The goodness of fit of the model is checked by the determination coefficient (R^2). The coefficient of determination (R^2) was calculated to be, 0.9858, 0.9675, 0.8627 and 0.9453 for WL, SG, RR and SS respectively. The R^2 value is always between 0 and 1, and a value >0.75 indicates aptness of the model. For a good statistical model, R^2 value should be close to 1.0. The value of CV is also low as 1.45, 4.43, 3.09, and 5.88 indicate that the deviations between experimental and predicted values are low. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. In this work the ratio is found to be > 7, which indicates an adequate signal.

Multiple regression equations were generated, relating responses in coded forms (levels) of process variables. The results of theoretically predicted response are shown in Table 2. The developed models, in the form of coded process variables, are as follows:

$$Y_1 = 39.32 + 1.90\ A + 2.39\ B + 2.90\ C - 0.43\ AB - 0.12\ AC - 0.78\ BC - 0.91\ A^2 - 0.33\ B^2 - 0.57\ C^2 \quad (4)$$

$$Y_2 = 10.45 + 1.56\ A + 1.36\ B + 0.92\ C + 0.19\ AB + 0.061\ AC + 0.83\ BC + 0.93\ A^2 + 0.64\ B^2 - 0.30\ C^2 \quad (5)$$

$$Y_3 = 2.99 - 0.10\ A - 0.12\ B - 0.055\ C + 0.12\ AB - 0.040\ AC - 0.093\ BC + 0.012\ A^2 - 0.10\ B^2 + 0.068\ C^2 \quad (6)$$

$$Y_4 = 6.60 + 1.09\ A + 0.44\ B + 0.90\ C + 0.35AB + 0.025AC + 0.23BC - 0.20\ A^2 + 0.50\ B^2 + 0.17\ C^2 \quad (7)$$

where Y_1, Y_2, Y_3, Y_4 are water loss (%), solid gain (%), rehydration ratio and sensory score respectively, and

A, B, and C are the coded values of the test variables time (min), temperature (^{0}C) and sucrose concentration (^{0}Brix) respectively.

Water Loss

Table 3 indicates that linear terms of all process variables have significant effects ($p< 0.05$) on water loss. The quadratic term of time and interaction term of 'temperature and concentration' have significant effects on water loss during osmotic dehydration. The relative magnitude of coefficients (Table3) indicates the maximum positive contribution of osmotic solution concentration followed by solution temperature and process duration. These results indicate an increased water loss with the rise of osmotic solution concentration, solution temperature and process duration , which is clearly depicted in Figure 1. The quadratic term of processing time and the interactions of BC have negative effect on water loss.

Solid Gain

The p-values (Table 4) indicate that all linear terms of process variables have significant effects on solid gain during osmotic dehydration. The quadratic terms of time and temperature, and interaction term of BC have significant effects on solute gain. The magnitude of coefficient indicates the maximum positive effect of process time, followed by temperature and concentration, which implies increase in solid gain with the growth of process variables (Figure 2). The quadratic terms of processing time and temperature has positive effect on solid gain. Further interactive effect of BC also has positive effect on solid gain during osmosis.

Rehydration Ratio

The magnitude of p and F values in Table 5 indicates that linear terms of time and temperature have significant effect on rehydration ratio of osmotically pretreated grapes while negative signs of coefficient values indicate that, with increase of time and temperature, there will be decrease in rehydration ratio (Figure 3). The interactive term of AB also have significant effect on rehydration ratio. AB interaction has positive effect on rehydration ratio of osmotically dehydrated grapes.

Sensory Score

All the linear terms have significant effect on the sensory score of rehydrated grapes ($p< 0.05$) at 5% level of significance (Table 6). The linear terms of all the variables show the positive effect on sensory score, which implies rise in sensory score with increase in process variables (Figure 4).The magnitude of coefficients of linear terms shows that the process duration has more pronounced effect on sensory score than temperature and concentration. The quadratic term of temperature also shows the significant effect on sensory score and has a positive effect on the sensory score of rehydrated grapes.

Optimum Conditions for Osmotic Dehydration

A graphical multi-response optimization technique has been adopted to determine the workable optimum conditions for the osmotic dehydration of grapes. The criterion for constraints optimization was maximum possible water loss, rehydration ratio and sensory score, and the lowest solid gain. In order to optimize the process conditions for the osmotic dehydration process by numerical optimization technique, equal importance was given to all parameters (osmotic solution concentration, process duration and solution temperature). Similarly the equal importance ratings were given to all responses. The maximum operation conditions for concentration, temperature, and process duration were 60^0 Brix sugar concentration, 36.92^{0}C temperature, and 160.57 minutes respectively. Corresponding to these optimum conditions, the predicted value for water loss was 40.54 (g/100 g initial sample), 10.06 solid gain (g/100 g initial sample), 3.05 rehydration ratio and 7.63 sensory score.

Conclusions

Response surface methodology was effective in the optimization of process parameters for the osmotic dehydration of grapes in osmotic aqueous solutions of sucrose having concentrations in the range 40-600 Brix, 35–550C temperature and process duration of 100–200 min. The regression equations obtained are suitable to optimize conditions for desired responses within the range of conditions applied to this study. Graphical techniques, in connection with response surface methodology, aided in locating optimum operation conditions, which were experimentally verified and proven to be adequately reproducible. Optimum conditions obtained by graphical optimization were temperature -36.920C, processi160.57 minutes and sucrose concentration - 600 Brix solution to achieve maximum water loss, rehydration ratio, and sensory score, and lower solute gain.

Tables

TABLE 1 THE LEVELS OF DIFFERENT PROCESS VARIABLES IN CODED AND UNCODED FORMS FOR OSMOTIC DEHYDRATION.

Coded levels	*Uncoded values of process variables*		
	Time (A, min)	*Temperature (B,⁰C)*	*Sucrose concentration (C,⁰Brix)*
-1	*100*	*35*	*40*
0	*150*	*45*	*50*
+1	*200*	*55*	*60*

TABLE 2 EXPERIMENTAL DESIGNS IN CODED FORMS OF PROCESS VARIABLES AND VALUES OF EXPERIMENTAL DATA

Coded process variables			*Responses*			
Time (A)	*Temp (B)*	*Conc (C)*	*Water loss(Y_1)*	*Solid gain (Y_2)*	*Rehy. Ratio(Y_3)*	*Score (Y_4)*
1	*0*	*-1*	*37.498*	*12.013*	*2.968*	*7*
-1	*0*	*1*	*38.416*	*10.014*	*3.261*	*6.1*
0	*-1*	*1*	*39.946*	*10.148*	*3.014*	*8*
1	*1*	*0*	*41.621*	*15.421*	*2.801*	*9*
-1	*0*	*-1*	*32.485*	*9.043*	*3.124*	*5*
0	*1*	*-1*	*38.459*	*9.765*	*3.092*	*6.1*
1	*-1*	*0*	*37.348*	*11.784*	*2.831*	*7.1*
0	*0*	*0*	*39.321*	*10.481*	*2.997*	*6.7*
1	*0*	*1*	*42.942*	*13.228*	*2.945*	*8.2*
-1	*1*	*0*	*39.659*	*11.872*	*2.742*	*6*
0	*-1*	*-1*	*32.461*	*9.236*	*3.104*	*6*
-1	*-1*	*0*	*33.682*	*8.998*	*3.243*	*5.5*
0	*0*	*0*	*39.299*	*10.394*	*2.991*	*6.5*
0	*0*	*0*	*39.326*	*10.493*	*2.993*	*6.7*
0	*0*	*0*	*39.311*	*10.394*	*2.999*	*6.6*
0	*1*	*1*	*42.812*	*14.012*	*2.631*	*9*
0	*0*	*0*	*39.346*	*10.497*	*2.990*	*6.5*

TABLE 3 ANALYSIS OF VARIANCE (ANOVA) FOR RESPONSE SURFACE QUADRATIC MODEL FOR WATER LOSS

Source	*Coefficient*	*Sum of*	*F- value*	*p-value*
Model	*-*	*150.87*	*53.82*	*< 0.0001*
Constant	*39.32*	*-*	*-*	*-*
A	*1.90*	*28.75*	*92.33*	*< 0.0001*
B	*2.39*	*45.67*	*146.63*	*< 0.0001*
C	*2.90*	*67.36*	*216.26*	*< 0.0001*
AB	*-0.43*	*0.73*	*2.33*	*0.1707**
AC	*-0.12*	*0.059*	*0.19*	*0.6757**
BC	*-0.78*	*2.45*	*7.87*	*0.0263**
A^2	*-0.91*	*3.51*	*11.29*	*0.0121**
B^2	*-0.33*	*0.46*	*1.47*	*0.2651**
C^2	*-0.57*	*1.38*	*4.42*	*0.0737**
Lack of Fit		*2.18*	*2355.83*	*< 0.0001*
R^2		*0.9858*		
Adj R^2		*0.9674*		
CV%		*1.45*		
Std. Dev		*0.56*		
Adeq Precision		*24.722*		

*Non – significant at 5% level of significance

TABLE 4 ANALYSIS OF VARIANCE (ANOVA) FOR RESPONSE SURFACE QUADRATIC MODEL FOR SOLID GAIN

Source	*Coefficient*	*Sum of squares*	*F- value*	*p-value*
Model	*-*	*49.95*	*23.13*	*0.0002*
Constant	*10.45*	*-*	*-*	*-*
A	*1.56*	*19.59*	*81.64*	*< 0.0001*
B	*1.36*	*14.86*	*61.94*	*<0.0001*
C	*0.92*	*6.74*	*28.10*	*0.0011*
AB	*0.19*	*0.15*	*0.61*	*0.4616**
AC	*0.061*	*0.015*	*0.062*	*0.8105**
BC	*0.83*	*2.78*	*11.59*	*0.0114*
A^2	*0.93*	*3.61*	*15.03*	*0.0061*
B^2	*0.64*	*1.73*	*7.22*	*0.0312*
C^2	*-0.30*	*0.39*	*1.61*	*0.2451**
Lack of Fit		*1.67*	*197.31*	*< 0.0001*
R^2		*0.9675*		
Adi R^2		*0.9256*		
CV%		*4.43*		
Std. Dev		*0.49*		
Adeq Precision		*17.261*		

*Non – significant at 5% level of significance

TABLE 5 ANALYSIS OF VARIANCE (ANOVA) FOR RESPONSE SURFACE QUADRATIC MODEL FOR REHYDRATION RATIO

Source	*Coefficient*	*Sum of squares*	*F- value*	*p-value*
Model	*-*	*0.37*	*4.89*	*0.0241*
Constant	*2.99*	*-*	*-*	*-*
A	*-0.10*	*0.085*	*10.02*	*0.0158*
B	*-0.12*	*0.11*	*12.63*	*0.0093*
C	*-0.055*	*0.024*	*2.81*	*0.1375**
AB	*0.12*	*0.055*	*6.53*	*0.0378*
AC	*-0.040*	*0.3186*	*0.75*	*0.4140**
BC	*-0.093*	*0.034*	*4.05*	*0.0839**
A^2	*0.012*	*0.1157*	*0.074*	*0.7929**
B^2	*-0.10*	*0.044*	*5.16*	*0.0573**
C^2	*0.068*	*0.020*	*2.31*	*0.1723**
Lack of Fit		*0.059*	*1319.12*	*< 0.0001*
R^2		*0.8627*		
Adi R^2		*0.6862*		
CV%		*3.09*		
Std. Dev		*0.092*		
Adeq Precision		*7.695*		

*Non – significant at 5% level of significance

TABLE 6 ANALYSIS OF VARIANCE (ANOVA) FOR RESPONSE SURFACE QUADRATIC MODEL FOR SENSORY SCORE

Source	*Coefficient*	*Sum of squares*	*F- value*	*p-value*
Model	*-*	*19.50*	*13.45*	*0.0012*
Constant	*6.60*	*-*	*-*	*-*
A	*1.09*	*9.46*	*58.74*	*0.0001*
B	*0.44*	*1.53*	*9.51*	*0.0177*
C	*0.90*	*6.48*	*40.23*	*0.0004*
AB	*0.35*	*0.49*	*3.04*	*0.1246*
AC	*0.025*	*0.1244*	*0.016*	*0.9044*
BC	*0.23*	*0.20*	*1.26*	*0.2992*
A^2	*-0.20*	*0.17*	*1.05*	*0.3405*
B^2	*0.50*	*1.05*	*6.54*	*0.0377*
C^2	*0.17*	*0.13*	*0.80*	*0.4006*
Lack of Fit		*1.09*	*36.25*	*0.0023*
R^2		*0.9453*		
Adj R^2		*0.8751*		
CV%		*5.88*		
Std. Dev		*0.40*		
Adeq Precision		*13.726*		

*Non – significant at 5% level of significance

Figures

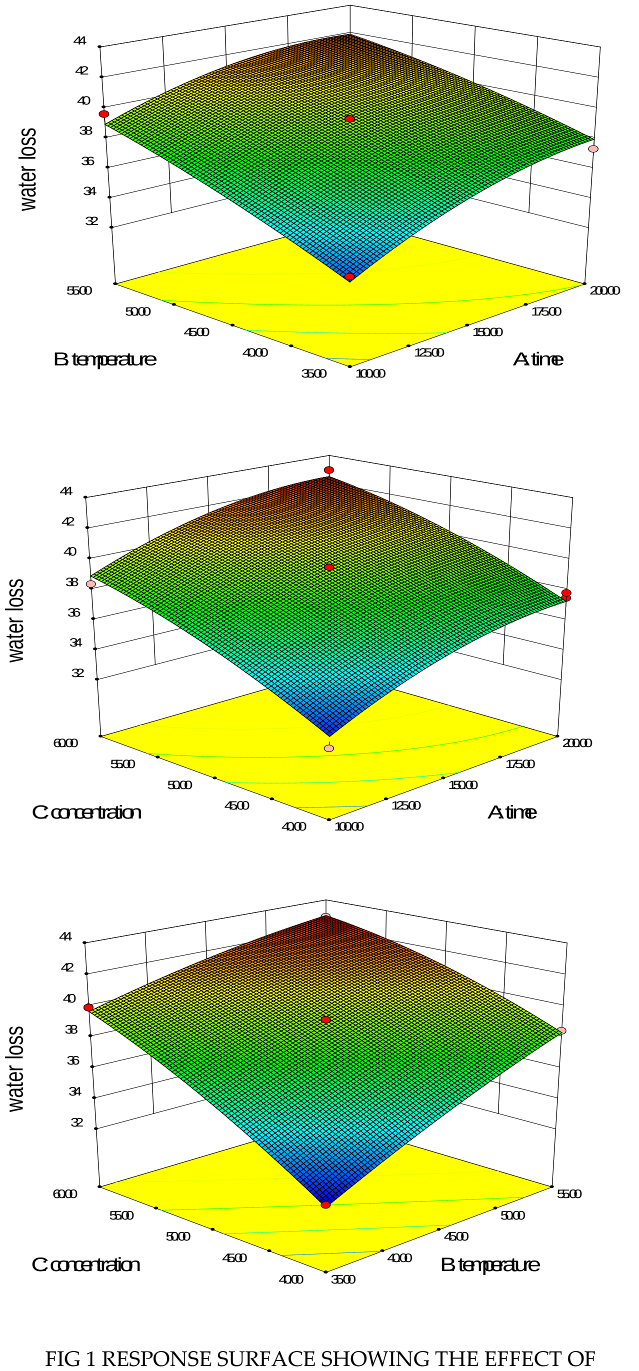

FIG 1 RESPONSE SURFACE SHOWING THE EFFECT OF VARIABLES (a) A AND B (b) A AND C (c) B AND C ON WATER LOSS DURING OSMOSIS

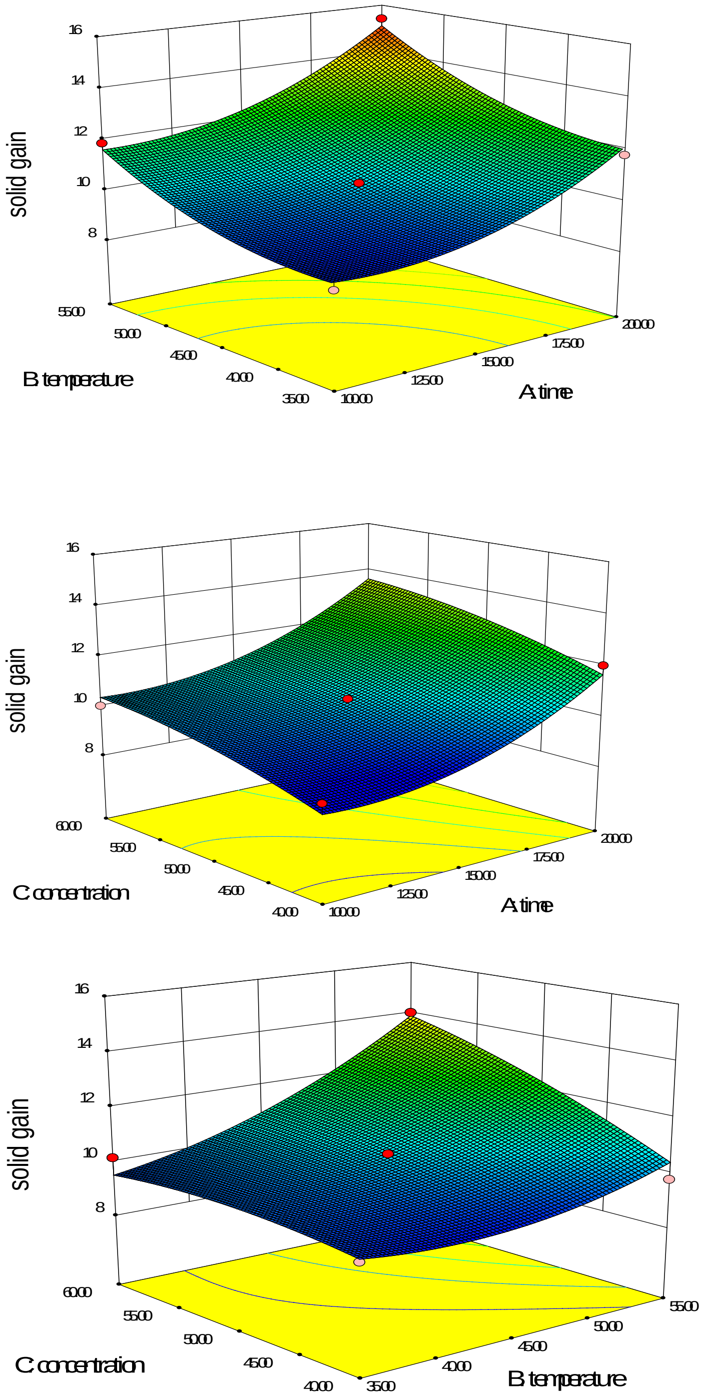

FIG 2 RESPONSE SURFACE SHOWING THE EFFECT OF VARIABLES (a) A AND B (b) A AND C (c) B AND C ON SOLID GAIN DURING OSMOSIS

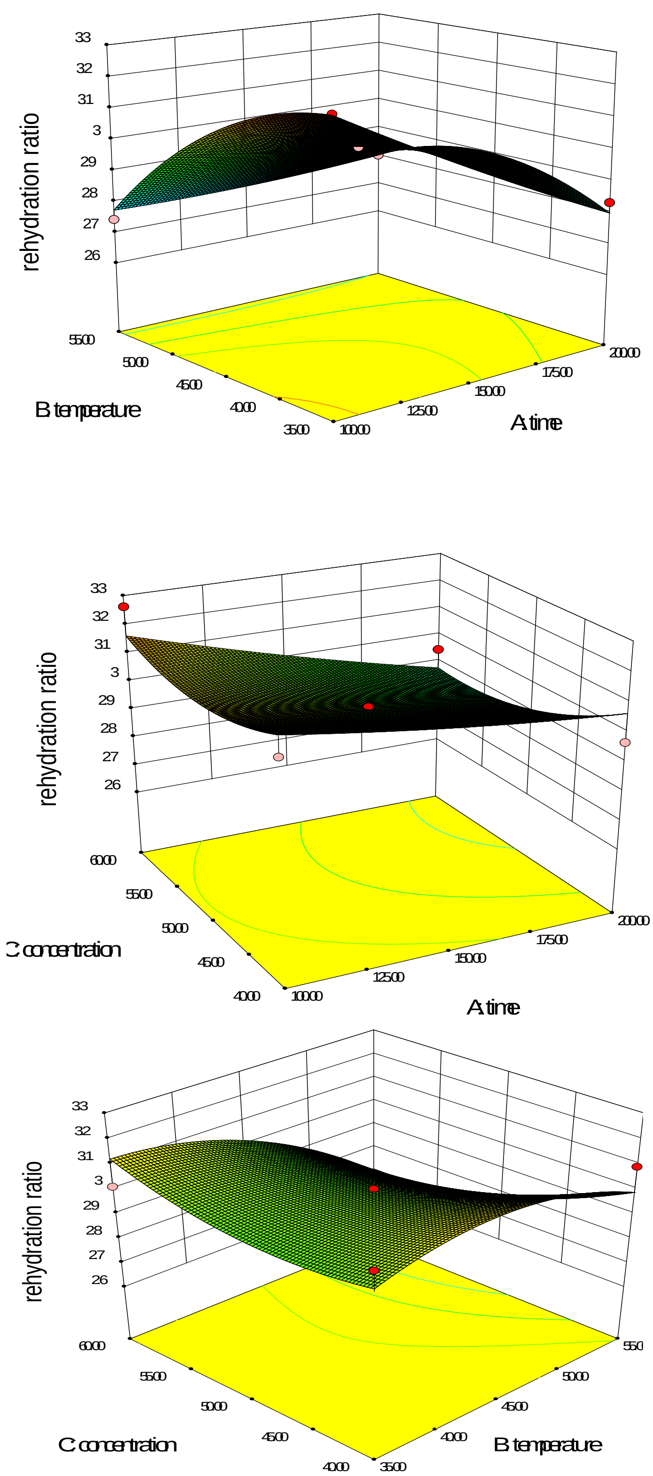

FIG. 3 RESPONSE SURFACE SHOWING THE EFFECT OF VARIABLES (a) A AND B (b) A AND C (c) B AND C ON REHYDRATION RATIO DURING OSMOSIS

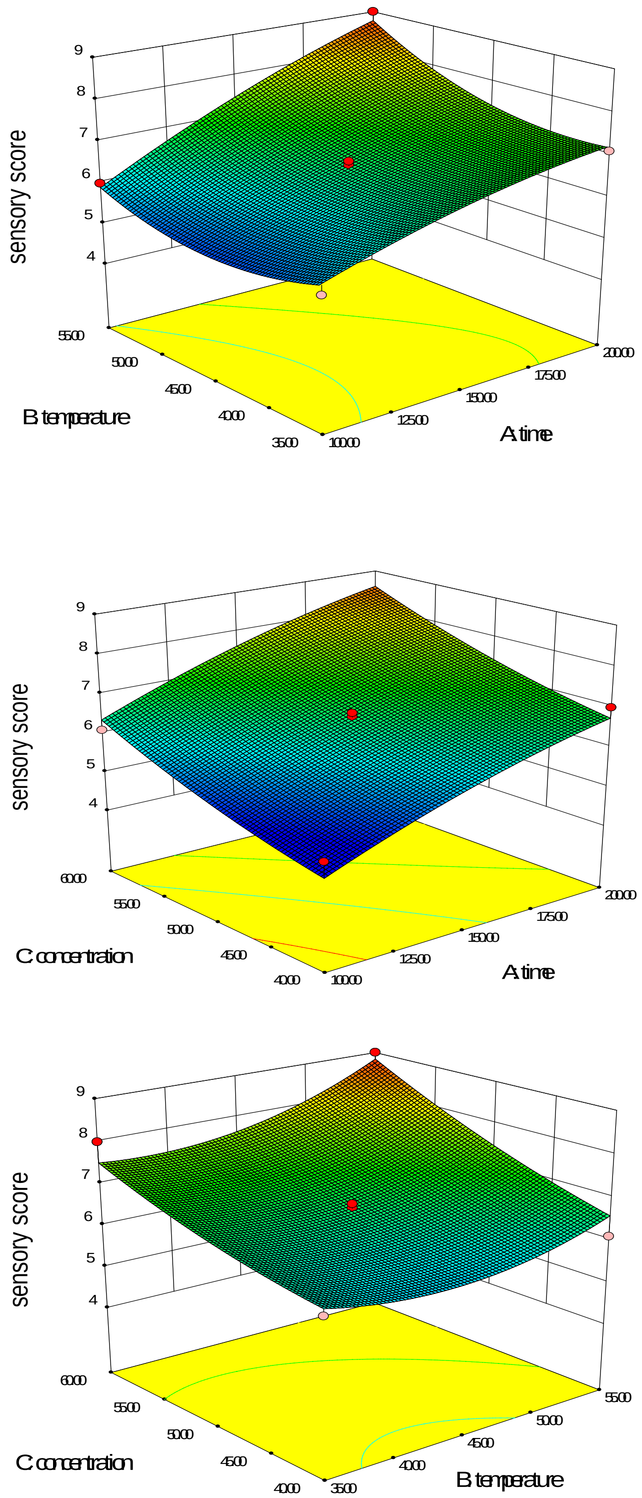

FIG .4 RESPONSE SURFACE SHOWING THE EFFECT OF VARIABLES (a) A AND B (b) A AND C (c) B AND C ON SENSORY SCORE DURING OSMOSIS

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